

REMARKS/ARGUMENTS

The amendment to Claim 77 is supported by this claim as previously presented. Other amendments to previously presented claims are conforming in nature. Support for new Claim 100 is found, e.g., at specification page 4, lines 30-31. No new matter has been entered.

The above amendment has focused the claims on a preferred embodiment where glycerol is subjected to a reaction with a chlorinating agent in the presence of a carboxylic acid catalyst selected from glutaric acid and adipic acid to produce dichloropropanol. This reaction is not suggested by the combination of DE '308 and Britton in view of Novelli and Satoshi, further in view of GB '633, as explained below.

The Office has recognized that neither DE '308 nor Britton use glutaric acid or adipic acid in a process for producing dichloropropanol by subjecting glycerol to a reaction with a chlorinating agent, as claimed. DE '308 discloses 1-2% of "an organic carboxylic acid," listing acetic acid, propionic acid, succinic acid and azelaic acids, among others, and shows acetic acid to be superior to the others. Britton identifies acetic acid and formic acid, and uses only acetic acid in the examples.

The Examiner has taken the position that the phrase "an organic carboxylic acid" in DE '308, and the use of the phrase "a catalyst such as formic or acetic acid" in Britton, is suggestive of other carboxylic acids not specifically mentioned in either reference, and that one of ordinary skill in the art would have been motivated by Novelli and Satoshi to use either a tricarboxylic acid (Novelli) or a C1 - C15 aliphatic mono- or polycarboxylic acid (Satoshi) in the DE '308/Britton process. See page 3, top, of the outstanding Official Action as well as page 5, paragraph 9 thereof.

Applicants disagree. First, the above amendment to the claims deletes carboxylic acid catalysts selected from tri- and tetra-carboxylic acids, thereby mooting the disclosure of Novelli. Second, and with regard to the possible use of one of Satoshi's mono- or

polycarboxylic acids in the DE '308/Britton process, and ignoring for the moment that Satoshi is limited to the chlorination of ether compounds that do not include glycerol, DE '308 clearly teaches those of ordinary skill in this art that dicarboxylic acids are less preferred than polycarboxylic acids, and, like Britton who only uses acetic acid, that the best catalyst is acetic acid.

Specifically, in examples 1 and 2 of DE '308 the yields of glycerol chlorohydrin ("monochlorohydrin") are lower for succinic and azelaic acids (i.e., dicarboxylic acids) than they are for acetic and propionic acids (i.e., monocarboxylic acids). This is particularly clear when comparing the acids at the same concentration of carboxylic acid moieties in the medium:

Acid	% (wt)	-COOH groups (moles)	Monochlorohydrin yield (% of theory)
Acetic (CH <sub>3</sub> COOH)	2	0.33	85
Succinic (HOOC(CH <sub>2</sub> ) <sub>2</sub> COOH)	2	0.34	72

In addition, in Example 3 of DE '308 a 96% yield is provided by acetic acid for the target mono- and di-chlorohydrins. Not coincidentally, many patent applications filed after DE '308 that focused on the same process made use of mono carboxylic acids like acetic acid (Britton and WO 2005/021476, of record) or formic acid (disclosed in Britton) and not di- or higher polycarboxylic acids. Consistent with this is Novelli's Abstract, which indicates that mono carboxylic acids are suitable for making dichloropropanol, while dicarboxylic acids are suitable for making monochloropropanediol.

Also consistent is Satoshi. While the Official Action recognizes that Satoshi does not deal with the chlorination of glycerol, as claimed, but instead is limited to ether alcohols,<sup>1</sup> to the extent considered relevant it must also be recognized that the reference shows, by comparing example 1 (acetic acid) and example 2 (adipic acid), that the latter is much less active even in this distinct reaction. Indeed, to obtain similar conversion and selectivities, adipic acid has to be used with a promoter (aluminum trichloride), and under harsher reaction conditions (autoclave under pressure).<sup>2</sup>

Applicants thus submit that a clear review of the disclosures of the applied references, when combined, do not fairly lead one of ordinary skill in the art to the presently claimed invention, where glycerol is subjected to a reaction with a chlorinating agent in the presence of a carboxylic acid catalyst selected from glutaric acid and adipic acid to produce dichloropropanol. To any extent the references are all combinable, they clearly do not teach the use of a dicarboxylic acid selected from glutaric acid and adipic acid for such purpose. As such, no *prima facie* case is presented.

In addition to the above, Applicants have compared their invention to processes much closer to the claims than anything actually attempted in the applied references by showing that unexpectedly better results are provided with adipic acid (a C6 dicarboxylic acid) and glutaric acid (a C5 dicarboxylic acid) than with succinic (C4) and azelaic acids (C9).

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<sup>1</sup> There is no evidence on the record that one of ordinary skill in the art would believe that the reaction conditions, reactants, etc. pertinent to the chlorination reaction of ethers, including alcohol ethers, would be transferrable to the chlorination reaction of glycerol.

<sup>2</sup> See the machine assisted translation of Satoshi attached hereto, examples 1 and 2 at pages 7ff, and page 6, line 14 (“Moreover as additives ... for reaction promotion ...”).

In particular, on September 13, 2010, Applicants filed the Declaration of Patrick Gilbeau, showing better dichloropropanol productivity and no loss of catalyst for the presently claimed invention, which Declaration stated that:

7. The superior results obtained for glutaric acid and adipic acids, dicarboxylic acids with 5 and 6 carbon atoms, could not have been expected from DE 197308 where it can be seen that succinic and azelaic acids, dicarboxylic acids with 4 and 9 carbon atoms show equivalent yields for the glycerol monochlorhydrin production (example 3).

Notably, and as explained in paragraphs 4., 5. and 6. of the Gilbeau Declaration, the tests conducted therein were carried out under identical conditions except of course for the acid type, and therefore the differences observed reflect only the impact of the acid on the chlorination reaction. Moreover, the Declaration explicitly states that the results are unexpected since they are better for dicarboxylic acids (glutaric and adipic) with a number of C atoms (5 and 6) intermediate between the number of C atoms in succinic (4) and azelaic (9) acids.<sup>3</sup> On the basis of DE '308, one would have expected the same performance.

Accordingly, and because GB '633 relates to the manufacture of epoxy resins, and has nothing to do with the chlorination of glycerol, Applicants respectfully submit that this case is now in condition for allowance as the combination of applied references does not suggest what Applicants' are claiming or the unexpected beneficial results that they have demonstrated for their claimed invention as compared to the closest prior art. Because even a combination of the disclosures of the several references applied against Applicants' claims fails to suggest a method as claimed herein where glutaric acid or adipic acid is used in a process for producing dichloropropanol by subjecting glycerol to a reaction with a

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<sup>3</sup> See, e.g., *In re Soni*, 54 F.3d 746, 34 USPQ2d 1684 (Fed. Cir. 1995) ("Mere improvement in properties does not always suffice to show unexpected results. In our view, however, when an applicant demonstrates *substantially* improved results, as Soni did here, and *states* that the results were unexpected, this should suffice to establish unexpected results *in the absence of* evidence to the contrary.")

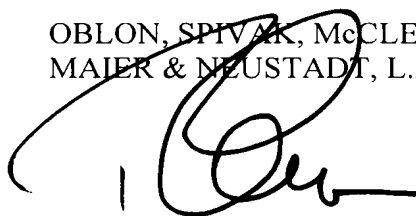
chlorinating agent, Applicants respectfully request the reconsideration and withdrawal of the rejection.

Finally, the provisional obviousness-type double patenting rejections are traversed as they have been made over later-filed applications U.S.S.N. 12/502,342 and U.S.S.N. 12/745,802, neither of which have been examined. In addition, the above Claims have been amended. Recognizing the fluid nature of the claims in each involved case pre-issuance, the MPEP directs that in such cases where double patenting is the only remaining issue "the examiner should withdraw that rejection and permit the earlier-filed application to issue as a patent without a terminal disclaimer." See MPEP 804. As the above amendments and the remarks place this case in condition for allowance, the provisional obviousness-type double patenting rejection should be removed herein and considered in the later-filed applications.

In view of the above, Applicants respectfully request an early Notice of Allowance.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, L.L.P.



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Richard L. Treanor  
Attorney of Record  
Registration No. 36,379

Customer Number  
**22850**

Tel: (703) 413-3000  
Fax: (703) 413 -2220  
(OSMMN 07/09)



## **MACHINE-ASSISTED TRANSLATION (MAT):**

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**(12)[GAZETTE CATEGORY]**

Unexamined Patent(A)

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(54) Title of the invention    Manufacturing method of chlorinated ether compound

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**(72)[INVENTOR]**

Kitamura Satoshi

5-23-6 Kuranamidai, Sodegaura-cho, Kimizu-gun, Chiba Prefecture

**(72)[INVENTOR]**

Uemura Shoji

5-7-1, Tatsumidai-higashi, Ichihara-shi

**(72)[INVENTOR]**

Tobita Shun

2-25-4, Sakuradai, Ichihara-shi

(71) Applicant Nisso Petrochemical Ind Co Ltd

4-8-15, Nihombashi Hon-cho, Chuo-ku, Tokyo

**[SPECIFICATION]****[TITLE OF THE INVENTION]**

The manufacturing method of a chlorinated ether compound

**[CLAIMS]**

1. When hydroxyl group of ether compound shown in following general formula

[I] is chlorinated using hydrogen chloride,

 $\text{RCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{OH}\dots[\text{I}]$ 

[R shows a hydroxyl group or a chlorine atom here.]

Carboxylic acid is made to exist.

The manufacturing method of the chlorinated ether compound shown in the following general formula [II] characterized by the above-mentioned.

 $\text{RCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{Cl}\dots[\text{II}]$ 

[R is the same as the above-mentioned here.]

2. The carboxylic acid made to exist is the 1 type, or 2 or more types of carboxylic acid chosen from the group which consists of C1-C15 aliphatic mono or poly carboxylic acid, C6-C15 ring group mono or poly carboxylic acid, C7-C15 aromatic mono or poly carboxylic acid and mono or poly carboxylic acid with a

heterocyclic ring.

The method of Claim 1.

3. Quantity of carboxylic acid made to exist is 0.1 to 10 weight% with respect to compound shown in said General formula [I].

The method of Claim 1 or Claim 2.

## **[DETAILED DESCRIPTION OF THE INVENTION]**

### **[INDUSTRIAL APPLICATION]**

This invention relates to the advantageous manufacturing method of chlorinated ethyl ether useful as an organic industrial source.

### **[PRIOR ART]**

In chlorinating an alcoholic hydroxyl group, the method of conventionally using a thionyl chloride, phosphorus trichloride, phosphorus pentachloride, a phosphorus oxychloride, etc. is utilized,

Chlorinating hydroxyl groups, such as alkylene glycol and the chloroalkyl oxy alcohol, using these methods is also known well.

However,

By such a method, the above chlorinating agents were expensive, the utilization factor of chlorine in this chlorinating agent was low, and also a fault which mentioned that it was generated by a large amount of waste materials after that toxicity and the danger on handling are large or reaction existed.

On the other hand, when chlorinating the hydroxyl group of diethylene glycol and manufacturing 2-(2'-chloro ethoxy) ethanol advantageously, the method of using hydrogen chloride as a chlorinating agent is indicated by Unexamined-Japanese-Patent No. 58-67638 and No. 58-109441 in order to avoid the above faults.

However,

With ether like diethylene glycol used for the former,

The tendency which ethylene glycol with difficult separation with the compound which a principal chain is cut and is made into the objective by hydrogen chloride byproduces in most quantity is remarkable,

The yield of 2-(2'-chloro ethoxy) ethanol made into the objective falls, and it is not preferable,



Furthermore, in the latter, since it is difficult to separate this byproduct glycol from 2-(2'-chloro ethoxy) ethanol, in order to purify this, it extracts using a specific extractant,

Furthermore, complicated operation which was mentioned distilled and purifying was unavoidable.

Furthermore, also when chlorinating this 2-(2'-chloro ethoxy) ethanol using hydrogen chloride and manufacturing di (2-chloroethyl) ether, ethylene glycol byproduces in the same manner to the above,

It was difficult to separate from 2-(2'-chloro ethoxy) ethanol aiming at this.

#### **[PROBLEM TO BE SOLVED BY THE INVENTION]**

The above point is considered,

When manufacturing specific chlorinated ethyl ether, even if it uses hydrogen chloride as this chlorinating agent, a by-product with difficult separation with the product made into an objective like ethylene glycol will decrease extremely,

Therefore, it inquired in order to acquire the method it becomes easy to purify a product.

#### **[MEANS TO SOLVE THE PROBLEM]**

Namely, with this invention,

When the hydroxyl group of the ether compound shown in the following general formula [I] is chlorinated using hydrogen chloride,



[R shows a hydroxyl group or a chlorine atom here.]

Carboxylic acid is made to exist.

It is a manufacturing method of the chlorinated ether compound shown in the following general formula [II] characterized by the above-mentioned.



[R is the same as the above-mentioned here.]

The compounds shown in said General formula [I] which is a raw material used in this invention are specifically diethylene glycol and 2-(2'-chloro ethoxy) ethanol,

These are chlorinated in accordance with a method of this invention, and the former becomes 2-(2'-chloro ethoxy) ethanol.

And the latter becomes di (2-chloroethyl) ether.

The chlorinating agent used in this invention is hydrogen chloride as mentioned above,

However,

By the time of use, they are a single-unit or aqueous solution,

If it puts in another way, whichever of the form of hydrochloric acid may be sufficient,

However,

The selectivity of the product which the direction used alone makes the objective improves,

It is preferable.

The carboxylic acid used by this invention is form mentioned above,

Although what kind of thing may be used as long as it has a -COOH group,

Preferably it is C1-C15 aliphatic mono or poly carboxylic acid, C6-C15 alicyclic mono or poly carboxylic acid, C7-C15 aromatic mono or poly carboxylic acid and the mono or poly carboxylic acid with a heterocyclic ring,

Aliphatic carboxylic acid, such as formic acid, an acetic acid, propionic acid, a butyric acid, valeric acid, a caproic acid, lauric acid, an oxalic acid, malonic acid, adipic acid, a sebacic acid, dodecanedioic acid, acrylic acid, a crotonic acid, as these examples.; alicyclic carboxylic acid, such as cyclohexanedicarboxylic acid and a cyclo dodecane monocarboxylic acid; aromatic carboxylic acid, such as a benzoic acid, toluic acid, a terephthalic acid, trimellitic acid, hydroxybenzoic acid, and naphthalene carboxylic acid, can be mentioned.

Such carboxylate residue could be substituted by a halogen atom, an alkyl group, an alkoxy group, a hydroxyl group, a mercapto group, aromatic group, a cyano group, etc.

Furthermore, as such carboxylic acid,

Amino acid; carboxylic acid with various heterocyclic rings, such as furan, a thiophene, a pyrrole, a pyridine are used preferably.

Furthermore, a polymer or carboxylic type various ion exchange resins with carboxylic acid partial chains, such as polyacrylic acid, derivatives activated remarkably, such as these acid anhydrides, a chloride, and ester, can also be used.

The usage-amount of said carboxylic acid or a derivative is 0.1 to 10 weight% with respect to the compound shown in the said General formula [I] that is a raw

material,  
Preferably it is 0.5 to 5 weight%.

Although there is no limitation of a method in particular when enforcing a method of this invention,

For example, the method of adding hydrogen chloride continuously or intermittently in the compound shown by the said General formula [I] formula, the continuous method which supplies the compound and the mixture of hydrogen chloride to a reaction system, etc. can be illustrated.

Moreover, if still more nearly necessary, it is possible to also make another additive or a catalyst exist in a nuclear reaction at a nuclear reaction system in addition to an inactive solvent or the said carboxylic acid.

As a solvent which can be used, various hydrocarbons, such as a halogenated hydrocarbon, hexane, a heptane, an octane, benzene, toluene, and xylene, can be illustrated.

Moreover, as additives other than carboxylic acid, metal halides, such as the Lewis' acid for reaction promotion, for example, aluminum, zinc, iron, a tin, titanium, antimony, and magnesium, etc. can be illustrated.

As for especially reaction temperature, 80-120 degreeC of 50-150 degreeC is preferable,

If it is 50 degrees C or less, reaction is very slow, and at the temperature over 150 degreeC, the byproduct of an impurity becomes remarkable,

Moreover, occurrence of carbide is also seen,

It is not both preferable.

There is no limitation in particular in the reaction pressure,

Although reaction in the following wide ranges is possible under pressure reduction to under pressure,

If reaction rate is considered, it is preferable to advance reaction under some pressurization,

Usually, below the pressure of 5 kg/cm<sup>2</sup>\*G is enough.

In a method of this invention, a chlorinating agent is hydrogen chloride as mentioned above,

You should change this usage-amount according to the embodiment of this method.

For example, when the method of adding hydrogen chloride to the mixture of the

compound shown in the said General formula [I], i.e., diethylene glycol, 2-(2'-chloro ethoxy) ethanol, and the above carboxylic acid is assumed,

You may add hydrogen chloride until these all hydroxyl-containing compounds are provided to reaction,

However,

When reaction is advanced not much too much, the byproduct of an ethylene chlorohydrin will consist of diethylene glycol remarkably by principal chain cutting,

It exists in the tendency for the byproduct of an ethane dichloride to consist of 2-(2'-chloro ethoxy) ethanol remarkably,

Therefore

The addition amount of hydrogen chloride should consider and determine the composition of a product and by-product (It utilizes as a useful compound by isolating this thing depending on the case.) made into the objective after reaction.

Since the adding speed, the reaction temperature, and the reaction pressure of hydrogen chloride also affect the said composition similarly, you should take into consideration and determine these by integrating.

If hydrogen chloride remains in the reaction mixture manufactured in accordance with the method of this invention in this way,

This is driven out under pressure reduction,

It is set as an ethylene chlorohydrin by addition of ethylene oxide etc.,

Or what is necessary is to isolate 2-(2'-chloro ethoxy) ethanol or di (2-chloroethyl) ether made into the objective, and just to set it as a product by the well-known methods, such as distillation, after neutralizing with a basic compound.

### **[EXAMPLES]**

Although an Example and a comparative example are given to the following and this invention is demonstrated to it in more detail, it is not limited to these.

In addition, unless otherwise limited, describing it as "part" in the following descriptions shows a weight part.

### **[EXAMPLE 1]**

106 parts of diethylene glycol and 1.2 parts of acetic acid are added to the glass flask of 200 part by volume,

The thermometer, the agitator, and the condenser were attached and stirring and the heating were done.

When the temperature in a flask reached 100 degreeC, hydrogen chloride gas was introduced for 5 hours, and was made to react at a rate of 9.6 parts per hour.

It cools to 80 degreeC after termination of reaction,

A system is made with pressure reduction,

30 minute hydrogen chloride gas was driven out at 100 mmHg.

The reaction liquid weight in this time is 136.1 parts,

This is analysed in the gas chromatograph,

Diethylene glycol

37.6vt%

2-(2'-chloro ethoxy) ethanol 28.2vt%

Ethylene chlorohydrin 17.6vt%

Di (2-chloroethyl) ether 3.5vt%

and

Ethylene glycol 0.3vt%

It is a composition of above.

The production of ethylene glycol was very small.

The conversion ratio of the diethylene glycol calculated from the said result is 51.7%,

The selectivity of ethylene chlorohydrin of the selectivity of 2-(2'-chloro ethoxy) ethanol was 28.7% 59.6%.

2.3 parts of sodium carbonate is added to this reaction liquid, and excess hydrogen chloride is neutralized,

It rectified using a Widmer-type rectifier 30 cm in length,

38.2 parts of 2-(2'-chloro ethoxy) ethanol of purity 99.3% and 0.5% of ethylene glycol content rate were obtained.

The yield with respect to the used diethylene glycol was 30.7%.

#### **[COMPARATIVE EXAMPLE 1]**

An acetic acid was not added but Example 1 was repeated.

This analyzed the reaction liquid,

The conversion ratio of diethylene glycol is 45.3%,

The selectivity of 2-(2'-chloro ethoxy) ethanol is 51.8%,  
And the selectivity of ethylene chlorohydrin was 28.2%.  
Furthermore, the content rate of ethylene glycol was as high as 3.2vt%.  
This rectified this reaction liquid by the same method as Example 1,  
Although 36.7 parts of 2-(2'-chloro ethoxy) ethanol were obtained, the content  
rate of ethylene glycol was as high as 2.9%.  
And the yield with respect to the used diethylene glycol was as low as 10.6%.

#### **[COMPARATIVE EXAMPLE 2]**

32 parts of diethylene glycol is put into the glass flask which has a thermometer,  
an agitator, a gas inlet tube, and a reflux tube, and 11 parts of hydrogen chloride  
gas was supplied and absorbed from the gas inlet tube, while cooling a liquid.  
Subsequently, it was made to react at 100 degreeC after temperature rising to  
100 degreeC for 5 hours.  
Then, it cooled and reaction was completed.  
The weight of this reaction liquid is 41 parts,  
This was analyzed by the gas chromatograph,  
Diethylene glycol 36.7vt%

2-(2'-chloro ethoxy) ethanol 26.4vt%  
Di (2-chloroethyl) ether 3.6vt%  
Ethylene glycol 3.0vt%  
Ethylene chlorohydrin 6.9vt%

It is the composition of the above,  
The by-product existed abundantly.  
The yield of 2-(2'-chloro ethoxy) ethanol in a reaction liquid was 28.9% (opposite  
diethylene glycol).

#### **[EXAMPLE 2]**

530 parts of diethylene glycol, 5.0 parts of aluminium chloride, and 5.5 parts of  
adipic acid are put into the glass-made autoclaves of 1000 part by volumes,  
It stirred and heated.  
Then, a pressure is reduced for system inside to 100 mm/Hg at 100 degreeC,  
and hydrogen chloride gas is rapidly introduced at the rate of 60 part / Hr,  
After making the reaction pressure into a normal pressure, it continues and

introduces so that it may become the maximum pressure  $1.0\text{kg/cm}^2\text{G}$ ,  
5 hours were required for introducing the hydrogen chloride gas of 159 parts of whole quantity.

After an introductory termination, the autoclave was opened and the reaction liquid was analyzed using the gas chromatograph,

The conversion ratio of diethylene glycol is 53.4%,

The selectivity of 2-(2'-chloro ethoxy) ethanol is 60.4%,

The selectivity of ethylene chlorohydrin is 27.8%.

And the content rate of ethylene glycol was 0.3vt%.

The reaction liquid was succeedingly distilled and purified after the dehydrochlorination under reduced pressure, and 199.7 parts (purity 99.1%) of 2-(2' chloro ethoxy) ethanol were obtained.

0.2% of ethylene glycol was contained in this, and there was almost no influence of the yield on the time of distillation.

The yield with respect to the diethylene glycol which this thing used was 32.0%.

### **[EXAMPLE 3]**

106 parts of diethylene glycol and 3.0 parts of benzoic acids are added to the glass flask of 300 part by volumes,

It adds while stirring 114 parts of 35% hydrochloric acid aqueous solution to this,  
It was made to react at 100 degreeC for 4 hours.

The reaction liquid was analyzed,

The conversion ratio of diethylene glycol is 35.6%,

The selectivity of 2-(2'-chloro ethoxy) ethanol is 52.6%,

The content rate of ethylene glycol was 0.5vt%.

The vacuum distillation of this reaction liquid was carried out, and 23.0 parts of 2-(2'-chloro ethoxy) ethanol of purity 99.2% were obtained.

The yield with respect to the diethylene glycol which this thing used was 18.5%.

### **[EXAMPLE 4]**

623 parts of 2-(2'-chloro ethoxy) ethanol, 6.5 parts of zinc chloride, and 10.0 parts of chloro-acetic acids are added to the same reactor as Example 2,  
It stirred and heated.

Then, a pressure is reduced for system inside to 100 mm/Hg at 100 degreeC,

Hydrogen chloride gas is rapidly introduced at the rate of 60 part / Hr,

After making the reaction pressure into a normal pressure, it continues and



introduces so that it may become the maximum pressure 3.0kg/cm<sup>2</sup>\*G,  
7 hours were required for introducing the hydrogen chloride gas of 150 parts of  
whole quantity.

The reaction liquid was similarly analyzed as Example 1,

The conversion ratio of 2-(2'-chloro ethoxy) ethanol is 46.8%,

The selectivity of di (2-chloroethyl) ether is 62.8%,

The selectivity of ethylene chlorohydrin is 25.4%,

And the selectivity of ethylene glycol was 0.2%.

#### [EXAMPLE 5-8]

106 parts of diethylene glycol is put into the same reactor as Example 1,

It reacts on the conditions shown in Table 2,

The obtained result was shown in Table 2.

第 1 表

実施例	カルボン酸 (部)	塩化水素ガス 導入量 (部)	反応条件 (温度×時間) (℃)	DEG* 転化率(%)	CEE** 転化率(%)	EG*** 転化率(%)
5	カプロン酸 2.3	71.4	110×10 (℃)	80.3	48.4	0.6
6	シクロヘキサノ ジカルボン酸 1.7	38.8	95×3 (℃)	47.6	61.3	0.2
7	ニコチン酸 1.2	41.7	100×4 (℃)	51.4	58.8	0.3
8	メルカプト プロピオン酸 1.1	42.3	90×5 (℃)	46.8	63.2	0.2

\*DEG : ジエチレングリコール、CEE : 2-(2'-クロロエトキシ)エタノール EG : エチレングリコール  
をしめす。



Table 1

Examples	Carboxylic acids (parts)	A	B	C	D	E
5	Caproic acid 2.3	71.4	110 x 10 (degC)	80.3	48.4	0.6
6	Cyclohexane Dicarboxylic acid 1.7	38.8	95 x 3 (degC)	47.6	61.3	0.2
7	Nicotinic acid 1.2	41.7	100 x 4 (degC)	51.4	58.8	0.3
8	Mercapto propionic acid 1.1	42.3	90 x 5 (degC)	46.8	63.2	0.2

A = HCl gas introduction amount (parts)

B = reaction conditions (temperature x hours)

C = DEG\* conversion degree (%)

D = CEE\*\* conversion degree (%)

E = EG\*\*\* conversion degree (%)

DEG\* = diethylene glycol

CEE\*\* = 2-(2'-chloroethoxy)ethanol

EG\*\*\* = ethylene glycol

**[ADVANTAGE OF THE INVENTION]**

A method of this invention is used,

Cutting of the principal chain of this ether decreases by having hydroxyl-containing chlorinated ether by hydrogen chloride, and chlorinating,

Therefore, production of the difficult by-product of separation with this ether decreases,

This ether with high purity is obtained with a sufficient yield.

**[PATENTEE/ASSIGNEE]**

Nisso Petrochemical Ind Co Ltd

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